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10 November 1999

SUBJECT: Authorization for Release of Technical Information, Control Number: AFRL-PR-ED-TP-1999-0215
Veselenak, J., "Successful R&D Leveraging using T2 Mechanisms: Dual Use Polyhedral Oligomeric
Silsesquioxane (POSS) Nanotechnology"
DoD Technology Transfer Integrated Planning Team Meeting (Statement A)

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ABSTRACT CLEARED 12 AUGUST 1999

Technical Abstract for DoD Technology Transfer Integrated Planning Team Meeting in Lake Tahoe, NV, Nov 16-18, 1999

AFRL-PR-ED-TP-FY99-062

Title: Military and Commercial Benefits of AFRL POSS Technology: Leveraging R&D Investments under Technology Transfer [This will be a slide presentation only.]

200-400 word abstract required by Army TTO, 12 Aug 99:

In an attempt to meet the U.S. Air Force's demand for a new generation of lighter weight, higher performance polymeric materials, the U.S. Air Force Office of Scientific Research and the U.S. Air Force Research Laboratory Propulsion Directorate have, for the past six years, pursued the development of new chemical feedstock technologies based on Polyhedral Oligomeric Silsesquioxanes (POSS).

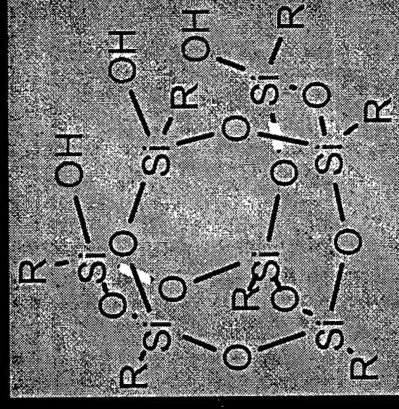
Within six years this investment has paid off with the development and large-scale production of the first new polymer feedstocks in the past forty years. POSS technology is also the only hybrid and nanostructured, silicon-based chemical feedstock technology developed to date. Because of its chemical nature (an inorganic core with organic side arms), POSS technology is easily incorporated into common plastics via copolymerization or blending and hence requires little or no alteration to existing manufacturing processes. POSS additives radically upgrade the thermal and physical properties of most plastics.

The Hybrid Polymer Team is composed of highly motivated technology champions from not only the Air Force, but industry and academia as well. The Directorate has formed relatively seamless strategic alliances with the University of Dayton Research Institute, Hybrid Plastics, LLC, University of California at Irvine, Michigan State University, and the University of Michigan. As a result, the Air Force in-house team has been able to bring in over a hundred thousand dollars per year of non-Air Force money from small and medium sized chemical companies through use of the Cooperative Research and Development Agreement mechanism. In fact, their fiscal year 2000 commercial reimbursement may exceed \$250,000. Thus, the directorate's core competency has been sustained even though the number of Air Force assigned personnel has declined in response to significant budget cuts to Air Force Propulsion R&D.

POSS partnerships have paid off in several respects. First, they have leveraged Air Force funds (6.1, New World Vistas, and 6.2) and DoD (Dual Use Science & Technology Program) funds with other government (the Commerce Department's Advanced Technology Program) and industry investment to help incorporate the technology into dual use applications. Second, the partnerships' developmental work has resulted in promising potential applications, including lower erosion rocket motor insulation; plastic rocket engine ducting; long duration, supersonic jet canopies; nanostructured lubricants; and atomic oxygen and ultraviolet resistant coatings. This network of partnerships, made possible under multiple Technology Transfer mechanisms, serves as a model for successful public-private R&D collaboration.

Successful R&D Leveraging using T2 Mechanisms: Dual Use Polyhedral Oligomeric Silsesquioxane (POSS) Nanotechnology

Jeff Veselenak
Manager, Technology Outreach Group
Propulsion Directorate
Air Force Research Laboratory

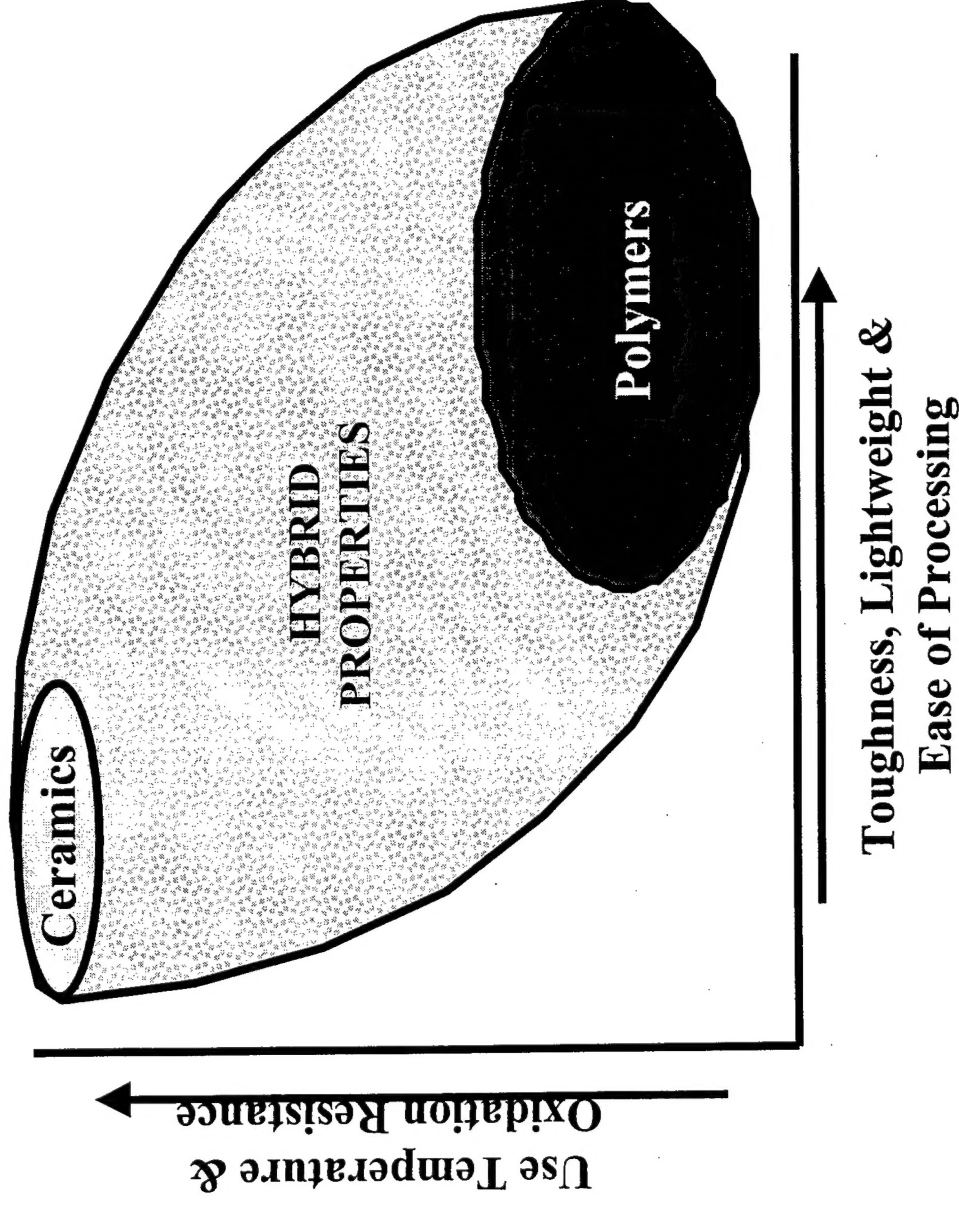


for the

DoD Technology Transfer Integrated Planning Team
Incline Village, Nevada
18 Nov 1999

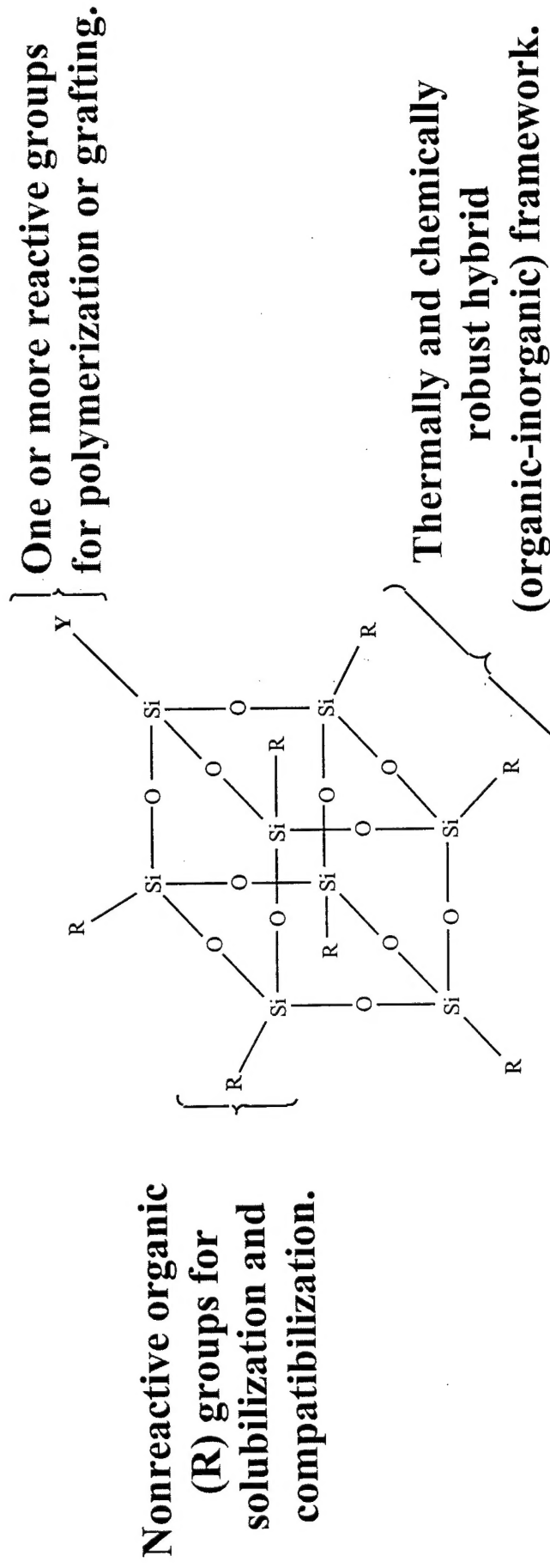
Propulsion (Air Force) Technology is Limited by Material Properties

Goal: Develop High Performance Polymers that REDEFINE material properties



• Hybrid plastics can bridge the barrier between ceramics and polymers

Anatomy of a POSS Molecule



Precise macromeric three-dimensional structure for molecular level reinforcement of polymer segments and coils.

Property Enhancements via POSS

Observed in POSS-Copolymers and Blends

increased T_g

increased T_{dec}

enhanced blend
miscibility

reduced
flammability

extended
temperature range

oxidation
resistance

reduced
heat evolution

increased
oxygen permeability

altered
mechanicals

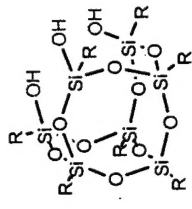
lower density

lower thermal
conductivity

reduced
viscosity

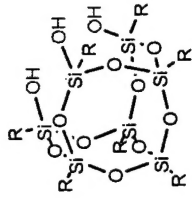
disposal
as silica

thermoplastic
or curable



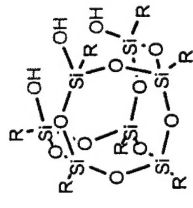
Events Facilitated by DoD T2

- 1996: Assignment of novel POSS structure and processing methods (patents) to UDRI
- 1996: Execution of a conduit (third party client) CRADA concept with UDRI--materials synthesis for reimbursement
- 1997: Polymer Working Group received AFOSR Star Team Award
- 1997: Significant increase of commercial interest in AFRL's POSS technology
- 1997: Inclusion of commercial funding into POSS R&D



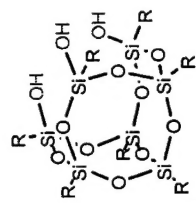
Events Facilitated by DoD T2

- 1998: Spin out of a commercial source (Hybrid Plastics, LLC (HP)) for bulk POSS monomer production
- 1998: Award of \$2M NIST ATP grant to HP
- 1998: Execution of a conduit CRADA between AFRL/PR and HP
- 1998: Tiger Team Assessment recommends WTN analysis
- 1999: WTN completes commercialization report
- 1999: Polymer Working Group and HP Team nominated for Gen Yates and FLC Awards



T2 Mechanisms Employed

- CRADAs - Hybrid Plastics, UDRI
- SBIR Contract - Maxdem (Polymeric Ducting and Housing)
- DUS&T TIA - Wright Materials Research (aircraft canopies and radomes)
- PIA - WTN (commercialization of POSS, supplying applied research contacts)
- MOA/MOU - JPL/NASA: the movement towards funding for space experiments



POTENTIAL MARKETS

- Rocket Propulsion Applications
- Air-Breathing Propulsion Applications
- Fire Resistant Materials
- Wire Insulation
- Contact Lenses
- Sporting Goods
- Blow Molded Films (Trash/Storage Bags)
- High Temperature epoxies and Resins
- Compounded Rubber
- Electronic Packaging
- Optical Plastics
- Dental Composites
- Structural Plastics
- R&D Chemicals

POSS Technology Development Timeline

Families of POSS-addition polymers

Families of POSS-condensation polymers

POSS
catalysis
UCI

4 POSS-Polymer
Architectures verified

POSS reagent
scale-up method
developed 0.5 kg +

International
interest

"Targeted"
resin

POSS
triol
GE

POSS monomer
polymerization
verified

POSS reagent
process
improvement

POMS
monomers
developed

development
"Synthetic /
processing"
advances

Technical Advances

1965 1989 1991 1992 1993 1994 1995 1996 1997 1998+

Milestones

POSS nanocluster
concept funded by
PL and AFOSR

1st POSS-monomer
tree completed
(catalog)

Non DoD
industrial
product
collaborations

Commercialization

DoD
product
collaborations

1st patent
issued

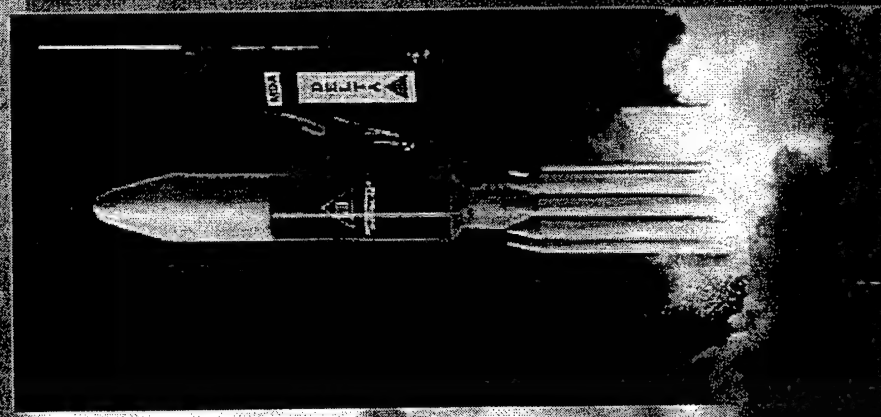
2nd patent
issued

3rd patent
issued

High-Performance Nanotechnology Materials

High-Performance Nanostructured Polymers

- Plastic tubing and ducting for liquid rockets engines
- High temperature case and motor insulation for solid rockets
- Space-resistant materials and coatings
- High-temperature canopies and hybrid lubricants

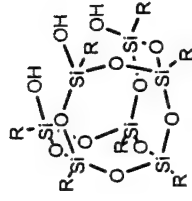


Nanostructured Polymers Offer Versatility!

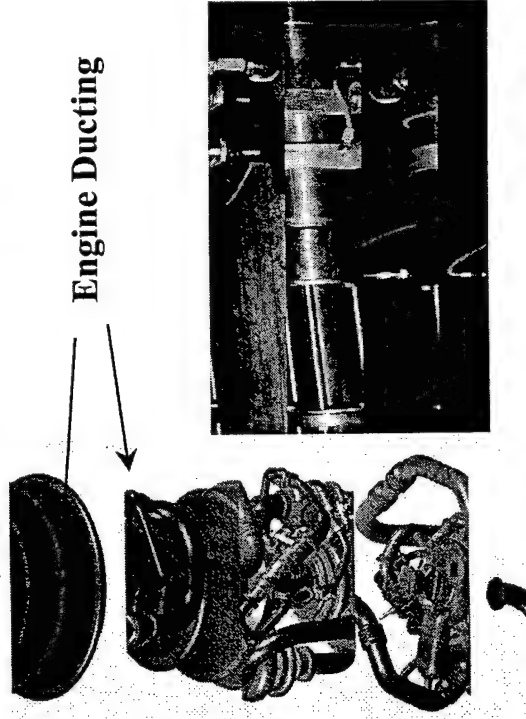
Take care
that these
pictures
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properly.

Plastics for Rockets

Crucial to Reducing Weight and Cost



Liquid Rocket Engines



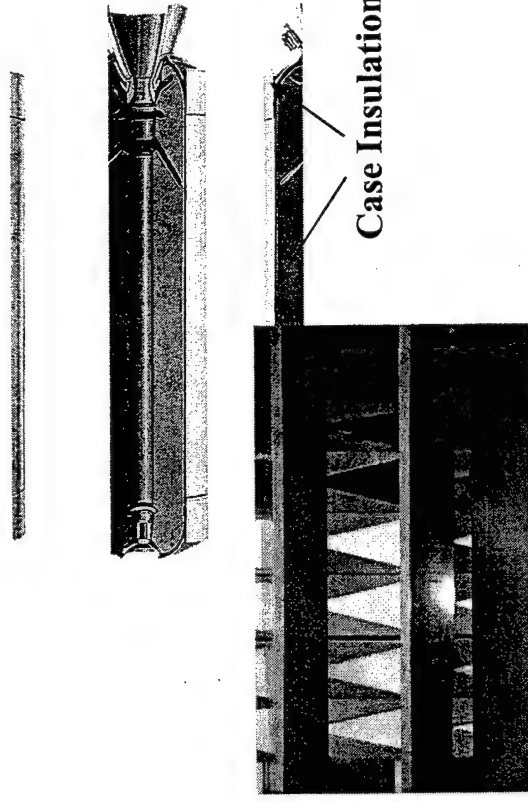
Polymer Tube/Case Hot Gas Burst Tester

Plastic Engine Ducting (SSME)

- 80% duct weight decrease
- 15% upper stage thrust-to-weight increase

3 candidates selected, SBIR, DUS&T

Solid Rocket Motors



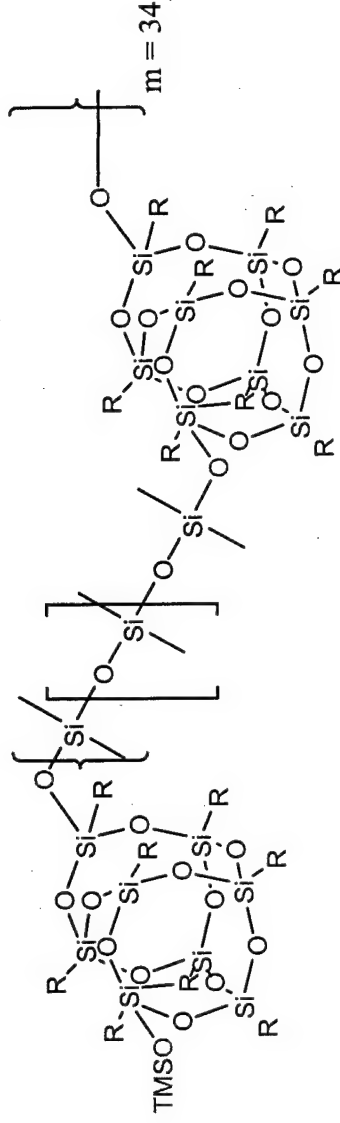
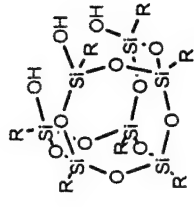
Char Motor Polymer Insulation Samples

50% Lower Erosion Insulation

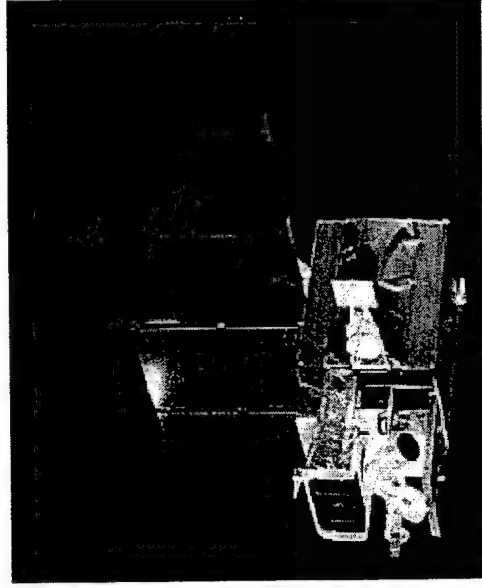
- Cuts Booster Insulation weight 44%
- Increases Booster Payload 7.4%

25% weight reduction & 6% density decrease
Current testing with motor mfr. (30 lbs. POSS!)

POSS Materials for Space Crucial to Reducing Weight and Cost



POSS-PDMS copolymers



Satellites & Space Systems

POSS Nanocomposite Payoffs

- **Maximum Space Resistance**
 - LEO, Atomic Oxygen (AO), VUV, Micro Impact
- **10% Lower Density**
- **High Modulus**
- **Resins for all Structural Applications**

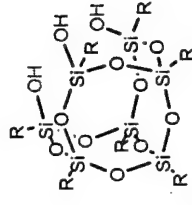
Simulated 3 mo. AO/VUV Exposure

- **10x greater AO resistance than current state of art**
- **Even better AO/VUV resistance**
- **Annealing of surface microcracks!!!**
- **Space-Inflatables (AFRL/ML)**

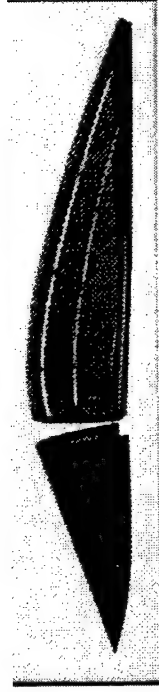
JPL collaboration, AO studies with Prof. Gar Hoflund, VUV with AFRL/ML

POSS Materials for Aerospace

High Temperature & Lightweight



Jet Canopies

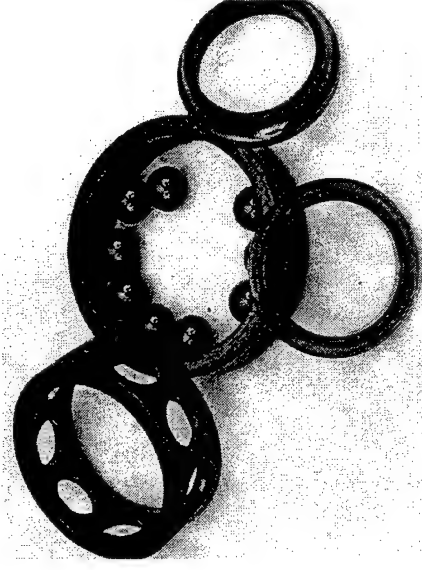


POSS-based Transparent Materials

- Mach 2.x speeds limited by plastic canopy (need increased HDT)
- Target Engagement Times can be reduced by increasing flight speed

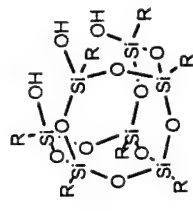
POSS-MMA increases use temp. by 150 °C
POSS-polycarbonate currently being prepared
Combining with nanocellular foam process
DUS&T with Wright Materials Research

Crucial Lubricant Applications



Nanostructured Lubricants

- Current lubricants limited to 400 °F
- POSS based lubricants $T_{dec} = 590$ °F
- Desire a fluid with working temperature range of -40° to 600° F (IHP/TET)



POSS Applications Summary

Addressing Propulsion Needs for High Performance Materials

- Lightweight, high-strength, high-temperature, & reduced cost
- Combine innovation with practicality
- Strong joint research effort with AFRL/ML-Materials Directorate
- Dual-use applications leveraged approach

AFRL/PR-West Research Group (+ Future)

Dr. Tim Haddad & Traudi Walker:

Basic Research - POSS size and R group effects
Applications - Jet Canopy, Radomes, Space

Dr. Rusty Blanski & Justin Leland:

Propulsion Applications - Lubes, Capacitors, Insulation
Basic Research - POSS blends and additives

Dr. Shawn Phillips & Dawn Hilton:

Applications - LRE ducting tubing, Insulation
Basic Research - high temp. polymers

Pat Ruth:

Basic Research - NWV Polymer Processing, blending
Applications - capacitors, insulation

Lt. Rene Gonzalez

Space-Resistant Materials, High-temp. polymers

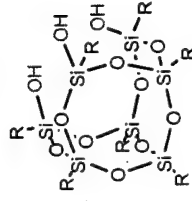
Synthetic Post-Doc:

Commercial - Customer X
Basic Research - R group effects
Applications - Case Insulation

2 Polymer Post-Docs + Assistant:

Commercial - Hybrid Plastics ATP, Customer X
Basic Research - R group effects
Applications - Insulation, Tubing and Ducting

AFRL Collaborations, Alliances, and Customers



POSS Monomers

Prof. Frank Feher*: UC Irvine, POSS molecule synthesis

Prof. Rick Laine*: U. of Mich., POSS molecule synthesis

Dr. Jim Spain: Tyndall AFB, POSS monomers via biocatalysis

Hybrid Plastics: CA, Supply of bulk POSS monomers for AF research

POSS Polymers

Dr. Pat Mather: AFRL/ML, POSSnorbornyl, POSSpolyurethanes

Dr. Rich Vaia: AFRL/ML, POSSparmax, POSS/Clay Comp.

Prof. Ben Hsiao*: New York U., POSSpolyurethanes

Prof. Andre Lee*: Mich. State U., POSSepoxy polymers

Dr. Jeff Gilman*: NIST, POSS ablative studies

Dr. Bill Wallace: NIST, Si-O-Si formation/opening

POSS Computational

Dr. Mark Gordon: Iowa State U., POSS formation, POSS polymers

Dr. Barry Farmer: AFRL/ML, POSS polymers

Funding: AFRL, AFOSR, other

*Directly Funded by AFRL/PRSM

Update
budget
Change
branch

Collaborations, Alliances, and Customers

Rocket Propulsion and Space Applications

2 Customers + JPL

POSS Lubes

AFRL/PRSL/F

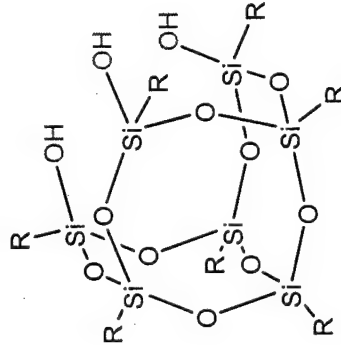
Fluorinated POSS

2 Customers

Hybrid Plastics

(40k FY99)

120 (180k FY00)



UDRI

(20k FY99)

(0K FY00)

Jet canopies/ Optical transparencies

Customer

Fire-safe Plastics

Customer/NIST/FAA

(60-150K FY00)

Commercial Sales

Aldrich/Gelest

WTN

Commercialization Support

Gas Separation

Customer

(60-120K FY00/01)

...and approximately 20 other proprietary customers.

Departing Champions

AFRL Labor Drain Associated with POSS
Monomer Production for Dual Use Applications
Prior to Spin Out of Hybrid Plastics, LLC

- 1 On site contracted PhD. - Full Time
- 1 DR-II (GS-12 equiv.) - Full Time
- 1 On site contracted technician - Full Time
- 1 DR-III (GS-14 PhD. equiv.) - 1/4 Time

Over \$300K per year in salaries

Hybrid Plastics' Key Strategic Alliances

Air Force Research Laboratory

Propulsion Sciences and
Advanced Concepts Directorate
Edwards Air Force Base, CA

Accelerate Technology

Small Business Development Center
Irvine, CA

University of Dayton

Research Institute

Special Programs &
Technology Commercialization
Dayton, OH

University of California

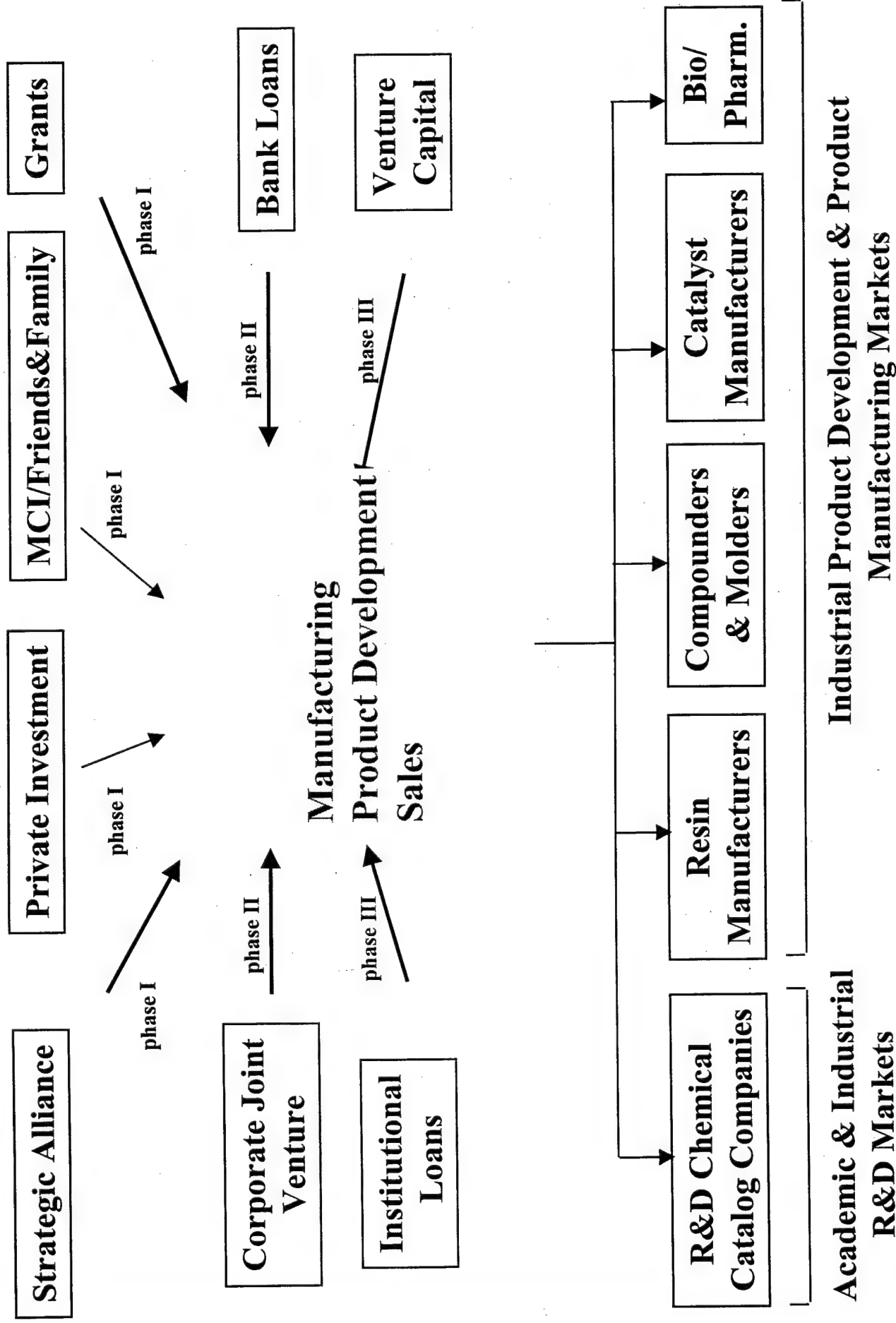
Irvine

Department of Chemistry &
Office of Technology Alliances
Irvine, CA

National Institute of Standards and Technology

Advanced Technology Program
Gaithersburg, MD

Hybrid Plastics' Business Spectrum



AFRL FY99-01 Polymer Research Goals

Polymer Synthesis/Characterization Studies

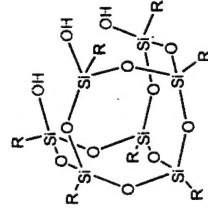
- **Reaction rates of POSS cages during copolymerization**
- **Type of POSS polymer formed (Bead vs. Pendant)**
- **Differences in size of POSS cages (Bead and Pendant)**
- **Varying non-reactive R groups (miscibility vs. agglomeration)**

Polymer Processing

- **POSS miscibility for blends (R group effect)**
- **Processing environment on polymer structure - how does this effect physical and mechanical properties?**
- **Innovative processing techniques**
- **Blend miscibility of two different POSS polymers**

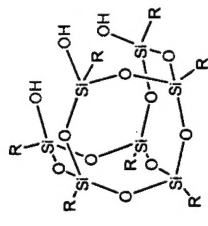
Polymer Studies Rely on Ability to Manipulate POSS Cages!!

The Future



Push AFRL/PR and AF POSS Applications:

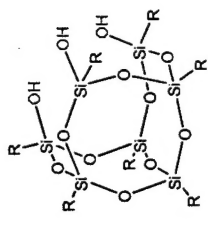
- Integrated High Payoff Rocket Propulsion Technology goals
- Synergy: Division (PRSL), Directorate (PRPE), AFRL (ML) DOD (Green Missile), NASA/JPL, DOC (NIST/FAA)
- Designed silicates (Clays, etc.,)
- Polymeric Cements (Geobond)
- Organometallic Polymers
- Functionally Graded Polymers (adhesion, property changes)
- Multi-Organometal Polymer Systems (smart systems)



What did AFRL gain?

- Increased 6.1/6.2 R&D leveraging using a conduit CRADA
- External customer funds (approx.):

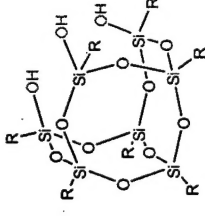
• FY97	\$80K
• FY98	50K
• FY99	90K
• FY00	est. 300K
- A commercial source of POSS monomer materials for research



What did AFRL gain?

- Reduced, via commercial outsourcing, the labor drain associated with:
 - POSS monomer production - a technology we already developed and patented
 - POSS marketing
- Sustained a DoD core competency/center of excellence using “out of the box” approaches

Lessons Learned from the Transfer of POSS Nanotechnology



- T2 champions are needed on both sides of the fence-a network of partnerships is best
- The champion S&Es should have an entrepreneurial interest in the advancement of the technology
- S&T funding cuts forced champions to think “out of the box” to advance the technology
- To gain trust of collaborators, the DoD must be diligent at protecting proprietary information-including its own
- The DoD must be aggressive with patenting its IP in hot fields